THE WASHING OF MANGANESE DIOXIDE AND CARBON MIXTURES OF OLD DRY BATTERIES

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Contents.

Introduction.

At the present time there are about fifty million dry batteries made per year which are used for various purposes, such as ignition for internal combustion engines, telephones, small lights, door bells, etc.

The dry battery consists of a zinc container lined with paper which is saturated with zinc chloride and ammonium chloride. In the center of the container a carbon rod is placed around which is tamped a mixture of manganese dioxide, carbon and graphite. The top of this mixture is then sprinkled with sand or saw-dust and then sealed with a layer of pitch. The composition of a dry battery is approximately:

Pyrolusite--85% MnO_2	100 parts.
Ground Coke	80 "
Artificial Graphite	20 "
Ammonium chloride	20 "
Zinc chloride-----30 degree Be.	7 "

The cost of materials for a # 6 battery, six inches high, is approximately 6.33 cents, while the factory labor is 1.5 cents making a total of 7.83 cents.

As will be seen from the analysis manganese dioxide, or pyrolusite, is one of the most important ingredients of a dry battery. Its purity for battery purposes should be at least 85 per cent available MnO_2. The most celebrated sources for this material are Ilmenau in Thuringia, Platten in Bohemia, Maehrisch-Trubau in Moravia. Other sources less notable are in the Caucasus, France, Spain, and North America. It occurs in the United States, abundantly in Vermont, and in Red

Island Bay at San Francisco. New Brunswick and Nova Scotia in Canada are also important sources.

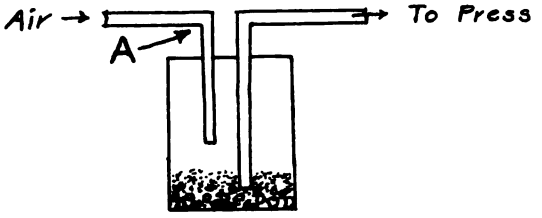
In view of the large number of dry batteries made each year and the slight depreciation of the manganese dioxide and carbon there is thus a basis for an interesting speculation of what becomes of the old cells. Since fifty per cent of the dry battery is made up of manganese dioxide it is at once evident that there is an enormous waste of this material.

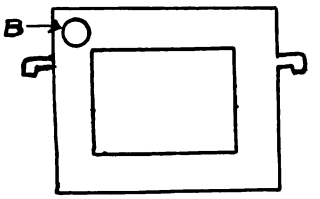
Due to the present conditions abroad the price of this material has risen enormously and importation has practically ceased.

The question therefore at once presents itself as to the feasibility of recovering the manganese dioxide from the oldworn out cells.which can be obtained at about twenty dollars a ton in various stages of corrosion.When obtained, the old batteries must be disintegrated so that the ingredients can be separated. The preparation of the pitch, carbon rods etc. for future use does not require a great deal of work. In order that the mixture of pyrolusite, graphite and carbon may be again used, it is necessary to wash out the zinc and ammonium salts contained therein.

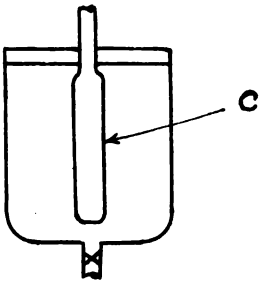
The problem then is the choice of a suitable filter press in which the zinc and ammonium salts can be easily removed. There is no information published concerning experimental data relating to material similar to that of dry batteries. At present a firm in Madison is beginning the recovery of old batteries, but so far no information concerning the results is available.

Various filter presses were tried in order to obtain one best adapted to the conditions. The Sperry press will not work due to the clogging up of the inlet holes,B,in the plates. Another source of trouble is with the mantejus. The material chokes in the bends of the pipes as at,A, and also settles out in the bottom of the press in layers.





The next type of filter press tried was a pressure tpye of Sweetland press. The manganese dioxide settled to the bottom while the graphite rose to the top. A film of graphite formed upon the filter leaf,C. This film is of very fine material and is practically impervious to the passage of water.



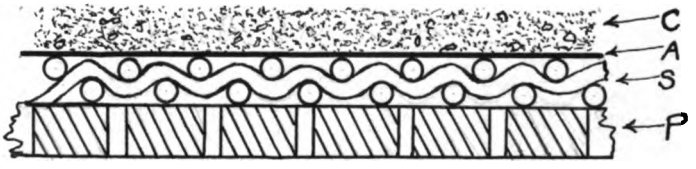
A centrifuge was next tried but was soon found to be of little value because there was a great loss of material. The fine carbon, graphite and also some manganese dioxide would pass through the screen due to the great centrifugal force. A large amount of water was also used for washing due to the fact that the water passed through the mass so quickly that it had very little time to dissolve out any zinc chloride.

It was next decided to use a pressure filter press.

The press consisted of two iron cylinders A and B Fig.1, between which was inserted an iron plate,C, which was perforated with one eighth inch holes. Two pipes enter through the cover, one for water, and one for air. A pressure gage was inserted in the air line so as to know at all times just what pressure was being used.

In the early investigations with this kind of press a canvas bag was inserted in the upper cylinder as a filtering medium. The material was placed directly in the bag. It was washed free from zinc chloride by filling the press with water and then applying air pressure. This method proved unsatisfactory due to the difficulty of keeping the bag tight against the walls of the iron cylinder.

The canvas bag was abandoned and instead of it a wire screen(S) Fig.2 was placed inside of the cylinder on top of the perforated plate. Over this screen a canvas cloth was tightly streched and the two cylinders A and B were then clamped together with the plate, wire and cloth between them. The reason for inserting a course wire screen between the steel plate and the canvas cloth was to allow a space between the two, thereby aiding filtration.



C-Filter Cake. S-Wire Screen.

P-Steel Plate. A-Filter Cloth.

Several difficulties became apparent after a few tests had been made. There was no way of telling just how much

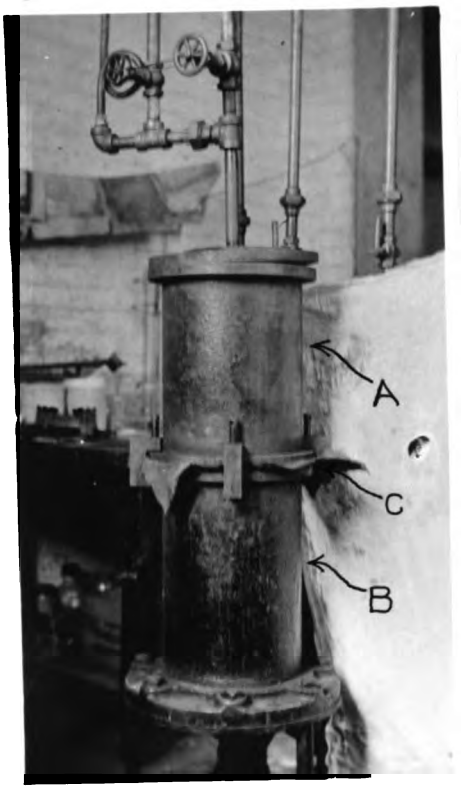


Fig. I

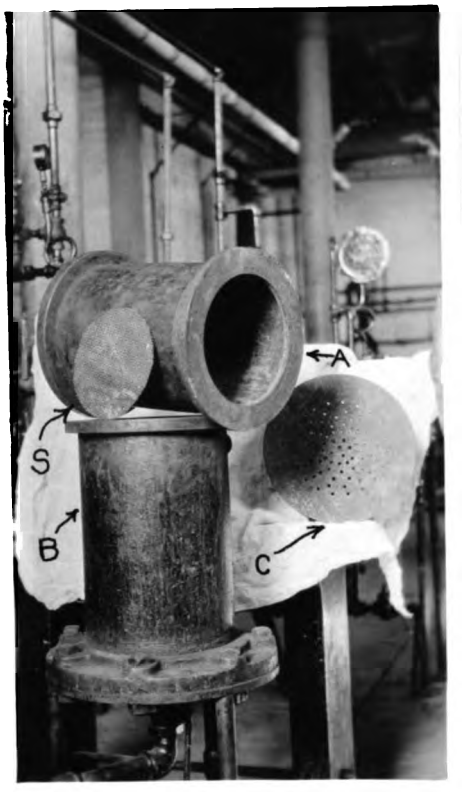


Fig. 2

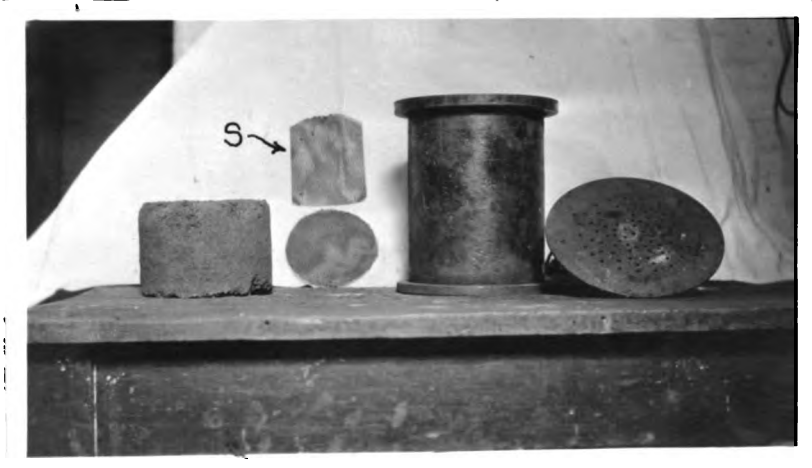


Fig 3

$$ZnCl_2 + 2\ NaOH = Na_2ZnO_2 + NaCl.$$

$$NH_4Cl + NaOH = NaCl + NH_4OH.$$

The digesting was continued until the evolution of ammonia vapors had ceased. The contents of the digester were then removed, leached for a day and filtered in the usual manner.

water was in the press at any time. If the press ran dry the air pressure would play directly upon the cake and compress it to a compact mass. As long as there is water in the press the cake was porous and allowed the water to pass through. This compact mass holds the water and causes a very slow filtration.

Another fault was the formation of holes in the center of the filter cake due to the stream of wash water entering through the top of the press and playing directly upon the cake. The wash water then passed through these holes instead of passing in a uniform sheet through the whole cake.

To avoid these difficulties, water pressure alone was used instead of air pressure. A wire screen(S) Fig.3 was placed directly under the water inlet pipe to break the stream and distribute the pressure evenly , thus eliminating the formation of holes. By making these changes no further trouble was experienced, as formerly, and good washing results are obtained.

After running several tests it was found that it was impossible to remove the last traces of zinc chloride no matter how long washing was continued? This is due to the formation of zinc oxychloride according to the following equation :

$$ZnCl_2 + H_2O = HCl + ZnOHCl.$$

In order to prevent the formation of this salt, the mixture of manganese dioxide and carbon is first treated with caustic soda in a digester . This results in the formation of sodium zincate and ammonia according to the following equations :

General Method of Obtaining Curves.

The zinc chloride was obtained by taking samples from every liter of wash water and from this sample analyzing an aliquot part as given under "Vol. of Sample" in the data. These values of zinc chloride per liter were plotted directly against the corresponding liter.

The time curve was plotted cumulative and needs no explanation.

The curve showing the per cent of washing of zinc chloride was obtained by calling the total amount of zinc chloride removed up to the point where the curve becomes horizontal, 100 per cent. Any intermediate point is then found by dividing the grams of zinc chloride per liter for that particular point by the total weight of chloride removed at the 100 per cent reading. For example: there are 1.06 grams of zinc chloride per liter at the 8th liter, dividing this by the total weight of zinc chloride, 78.07, we get 1.479 per cent for the answer.

The caustic soda curve is obtained in a similar manner to the zinc chloride curve and needs no explanation.

Method of Analysis.

An analysis for chlorides was made in all tests that were run.

The samples were taken as indicated on the data sheets and further described under the method of procedure.

The analysis for chlorine was a simple titration with N/10 $AgNO_3$ using potassium chromate as an indicator. The end point was determined by the yellow color of the solution.

The chlorine was figured on the basis of $ZnCl_2$ in accordance with the following equation:

$$2\ AgNO_3 + ZnCl_2 = 2\ AgCl + Zn(NO_3)_2 .$$

Calculations are as follows:

N/10 $AgNO_3$ = 17 grams $AgNO_3$/liter.

1 c.c. of N/10 $AgNO_3 = [(136.3 \times 17) \div 170 \times 2] \times 1000 = 0.00681$ grams of ZnCl .

From Test # 1, sample # 1,

$34 \times 0.00681 \times 10 = 46.4$ grams of $ZnCl_2$/liter.

In Tests # 11 and 12, two analysis were made, one for alkali and one for chlorine. The analysis for alkali was a direct titration with 0.75 N H_2SO_4 using methyl orange as an indicator.

1 c.c. of 0.75 N $H_2SO_4 = 0.030$ grams of NaOH.

From run #11, sample # 1, the alkali in a liter sample is, $2.1 \times 0.03 \times 100 = 0.063$ grams.

In testing for chlorine the sample was first neutralized with sulphuric acid and then analyzed as stated above and figured on the basis of sodium chloride as follows:

$$AgNO_3 + NaCl = NaNO_3 + AgCl .$$

1 c.c. of $AgNO_3$ = 0.005853 grams of NaCl as made up.

Taking values from test # 11, sample # 1 the calculations are as follows:

0.00583×100×34.6 = 20.23 grams of NaCl per liter.

Discussion of Curves and Results.

Test # 1.

The data for this is given in Table# 1 and the curves plotted from this data are those on Plate # 1.

The most interesting fact brought out by this set of is that at the ninth liter of wash water the zinc chloride curve becomes parallel with the horizontal axis. The percentage curve emphasizes this phenomenon still further and shows that 98 per cent of the total zinc chloride removed in this test was washed out by the time the ninth liter of water had passed.

Washing might be discontinued after the fourteenth liter as the amount of zinc chloride per liter was only 0.18 of a gram.

Test # 2.

In this test the precipitate was about 1 inch thick.

The irregularities in the curves obtained for this test may be attributed to the fact that the wash water entering the press formed a hole about one inch in diameter in the center of the cake. Most of the wash water passed through this hole giving an uneven distribution of washing.

Further, the cell used in this test was a very old one. The manganese dioxide and carbon were in the form of lumps which retained the chloride tenaciously. As the lumps dissolved from time to time the entrained chloride was liberated thus giving irregular results.

Test # 3.

The aim of this test was to obtain information on the

efficiency of washing when the manganese dioxide and carbon introduced into the press are well mixed and free from lumps and also to obtain some information regarding the time of washing.

The thickness of cake was one half inch.

The curves for this run, shown in Plate # 3, are fairly regular with the exception of the zinc chloride curve between the third and sixth liter.

As explained under the heading of "Method", after the manganese dioxide and carbon was placed in the press, water was introduced until the press was full. The water was then shut off and air pressure used until all the water had filtered through the cake. The press was again filled with water and the process continued. It is evident that an appreciable amount of time is necessary to cut off the air when the press is allowed to run dry, and to turn on the water, properly regulated. Thus more time is given for the water to leach out the zinc chloride. This is born out by the fact that the zinc chloride in the sixth liter is higher than that in the fifth or seventh.

The time of flow for each liter of wash water after the press had run dry also increased considerably. This was due to the fact that the cake was compressed by the air pressure.

Test # 4.

In this test an endeavor was made to keep some water in the press at all times. The thickness of cake was approximately seven eights of an inch. The water cock was open

just a little to allow the water to trickle through continuously. There was no way of telling,however, how much water was in the press and it ran dry again.

Due to this difficulty the results obtained were much like those of the preceeding test.

Test # 5.

The thickness of cake was approximately one and one quarter inches. The test was run in order to have a series of curves which would show the effect of uniform variation in the thickness of the cake. Again there is a higher value of zinc chloride at the seventh liter than at the sixth and eighth.

At the twenty-second liter water pressure of 85 pounds was used so there is a sharp turning point from the 45 pound pressure to the final pressure.

In spite of the fact that the curves have a number of irregularities in them, they all show that there is a tendency for the zinc chloride curve to become parallel with the horizontal axis.

Test # 6.

This test was run in order to ascertain as to whether leaching the mixture previously to introducing it in the filter press would increase the efficiency of washing.

The manganese dioxide and carbon mixture of fifteen batteries of various sizes, some of which were in good condition and others badly corroded, were dumped into a tank holding about fifteen gallons of water and were leached for one day.

The water was then drawn off and part of the mixture was transferred to the filter press giving a cake about two inches thick. Water pressure alone was used thus eliminating any danger of the press running dry.

The curves obtained were more regular than any of the preceeding ones and the content of zinc chloride per liter, as the washing continued, was much lower than any of the previous tests.

The zinc chloride curve becomes almost horizontal at the eleventh liter of wash water and washing can be stopped at this point.

Test # 7.

The object of this test was to obtain a check on test # 6 and to obtain some data on the time of washing.

The zinc chloride curve and the percentage curve are very much like those of Test # 6. As before, washing may be stopped with the eleventh liter.

One thing that is of great importance is the time of washing when using water pressure. In Test # 3 and # 4 the time per liter varied from fifteen seconds to one minute while in this test it averages about thirty seconds. This is therefore a great improvement over the preceeding tests.

Test # 8.

In this test the thickness of the cake was three and one half inches.

The curves obtained are again much like those of Tests 6 and 7. It will be noted that the chloride curve when parallel to the horizontal axis shows a greater content of zinc chloride

per liter than the other two.

Test # 9.

The data was obtained in the usual manner.

There is a slight irregularity in the chloride curve due to the fact that the press leaked and the run had to be stopped occasionally to patch up the leaks. Water pressure could not be applied until after the sixth liter due to this trouble.

The zinc chloride curve again seems to bring out the fact that the thickness of cake affects the washing results. There is a greater resistance to the flow of the water and hence the chloride does not come out as fast as where the cake is thinner.

In this test the eleventh liter contains 1.49 grams of zinc chloride as compared to 0.213 and 0.746 grams in the curves of Tests 7 and 8.

Test # 10.

This test was run to obtain results when the time of leaching was increased from one to two days and with a cake six and one quarter inches thick.

The results obtained in this test are the best throughout this investigation.

The curves are very regular and smooth and the zinc chloride in the eleventh liter is only 0.887 grams.

Test # 11.

This represents a run in which the material was digested with sodium hydroxide, leached for two days and then run in the regular manner.

The chloride and caustic soda curves are regular and need no explanation. Washing can be stopped at the tenth liter just as in the previous tests.

The important thing about this test is the length of run, the average per liter being about five and a half minutes.

This is probably due to the action of the alkali upon the cake. There was a thin layer, about 1/8 inch, of fine material on the top of the cake, and under this the regular coarse material.

This slimy, fine layer was evidently formed by the action of the caustic soda upon the manganese dioxide.

Test # 12.

In this test the mixture was leached before running in the digester and then again after it was digested. The mixture was then placed in the press and run in the regular manner.

The results are about the same. The time of flow per liter has shortened to an average of four minutes and the chloride content per liter is much lower at any given liter than in Test # 11.

The slimy layer was noticed in this case also.

Test # 1.

Two # 6 batteries were used.

Thickness of cake was not taken.

Air pressure of 45 pounds was used.

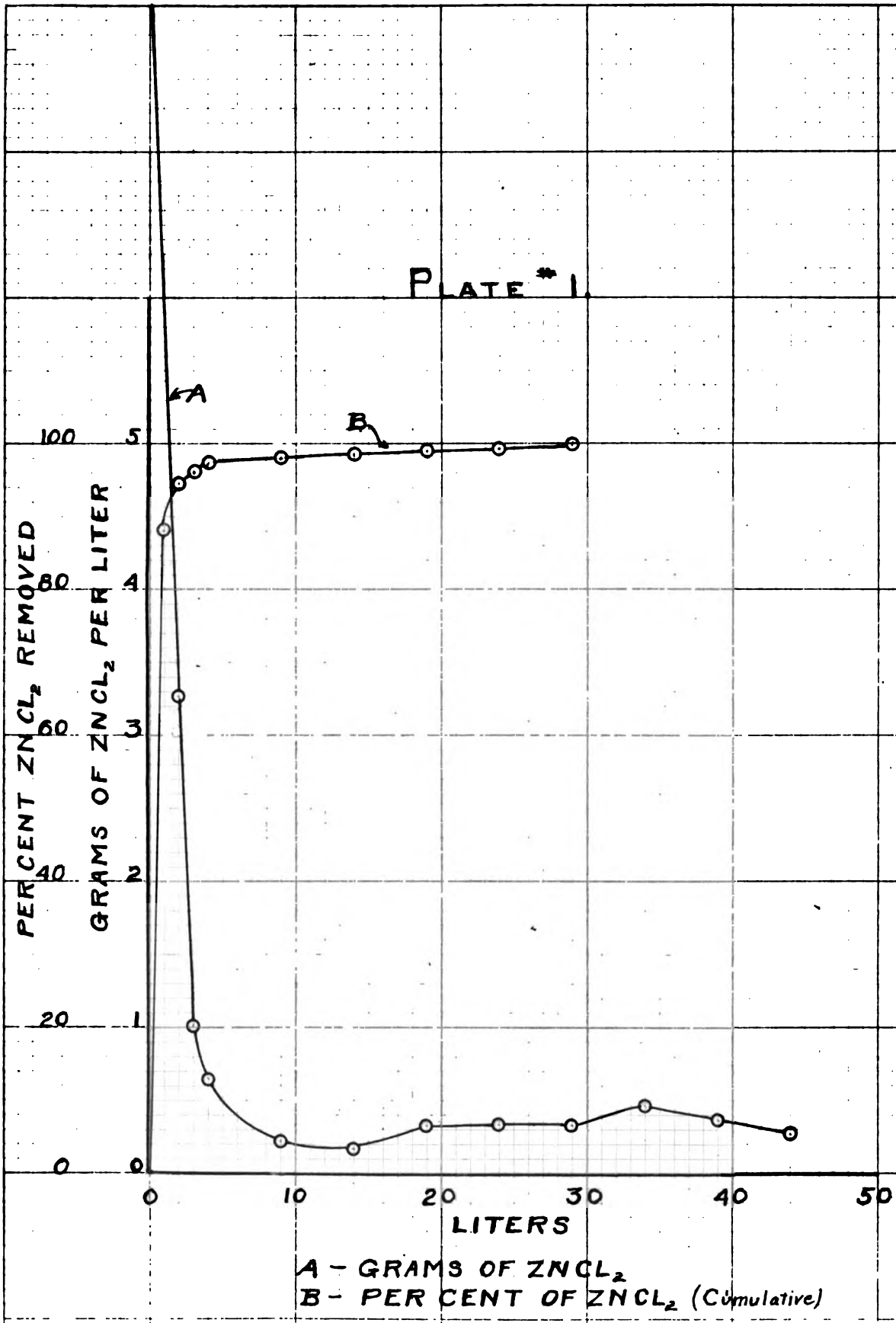
No time was taken.

Liter samples up to # 4. From # 5 on samples represent 5 liters.

Sample	Vol. of Sample	c.c. of N/10 $AgNO_3$	Gms. of $ZnCl_2$ /liter.	% of $ZnCl_2$.
1	50 in 500	34	46.4	88.3
2	50 in 1000	1.2	3.27	6.22
3	50 in 500	5.0	1.02	1.94
4	"	3.1	0.634	1.21
5	"	1.1	0.225	0.428
6	"	0.9	0.184	0.350
7	50 in 250	0.5	0.341	0.65
8	"	0.5	0.341	0.65
9	"	0.5	0.341	0.65
10	50 in 100	1.7	0.462	0.88
11	"	1.3	0.354	(a)
12	"	1.00	0.272	
13	"	1.0	0.272	
14	"	0.9	0.245	

(a) Total to here was called one hundred per cent.

PLATE # 1.



A - GRAMS OF $ZNCL_2$
B - PER CENT OF $ZNCL_2$ (Cumulative)

Test # 2.

One # 8 battery was used.

Thickness of cake was not taken.

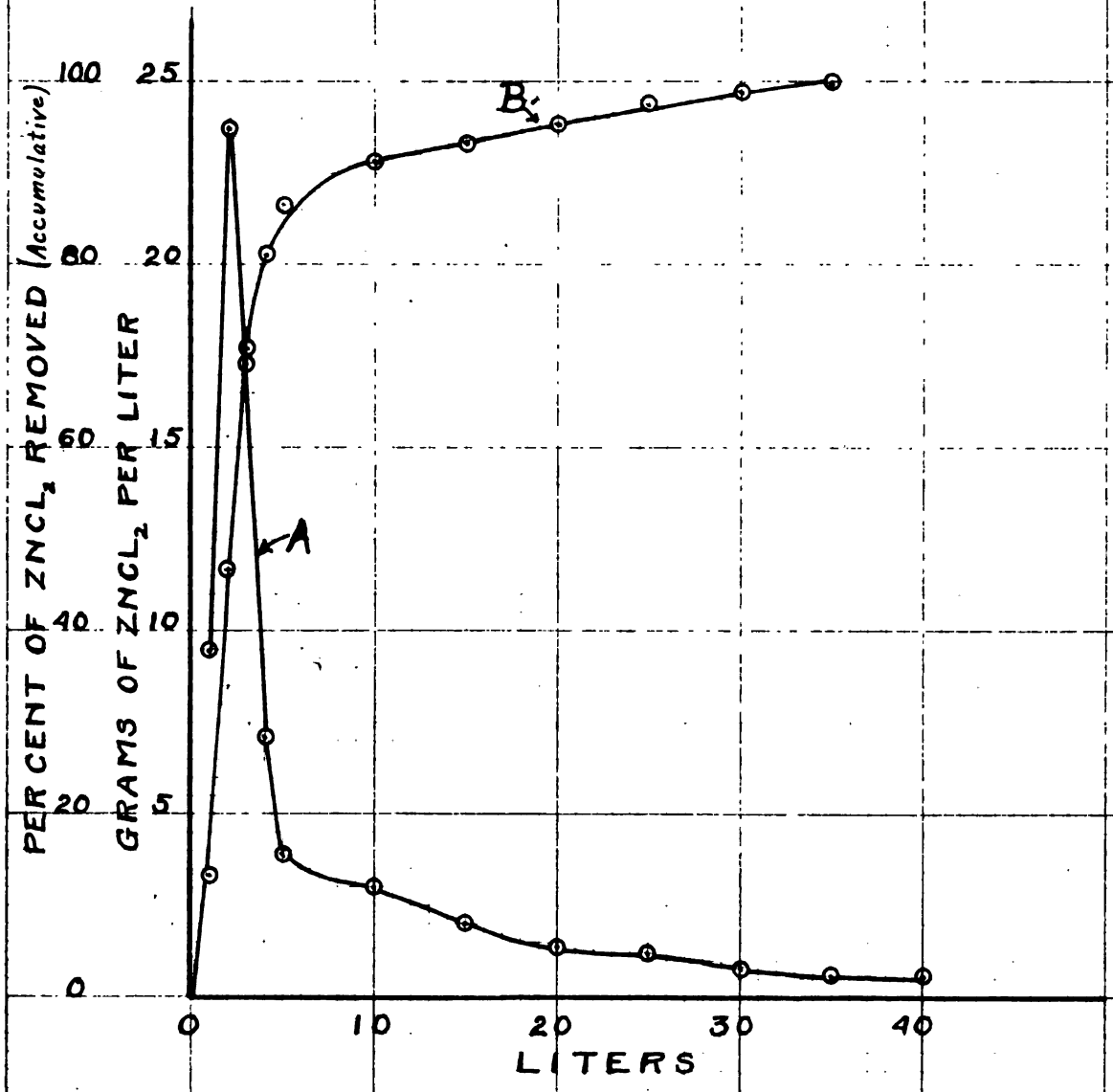
Air pressure of 45 pounds was used.

No time was taken.

Liter samples up to # 5.From #6 on samples represent 5 liters.

Sample	Vol. of Sample.	c.c.of $AgNO_3$.	Gms. of $ZnCl_2$/liter.	% of $ZnCl_2$.
1	50 in 500	6.9	9.45	13.35
2	"	17.4	23.7	33.40
3	"	12.7	17.35	24.40
4	"	5.2	7.1	10.00
5	"	2.9	3.95	5.55
6	"	2.2	3.00	4.22
7	"	1.5	2.04	2.87
8	"	1.00	1.36	1.92
9	"	0.9	1.22	1.72
10	"	0.6	0.82	1.15
11	"	0.4	0.545	0.768
12	"	0.4	0.545	0.768

PLATE # 2



A- GRAMS OF $ZNCL_2$
B- PER CENT OF $ZNCL_2$

Test # 3.

One # 6 battery was used.

Thickness of cake was not taken.

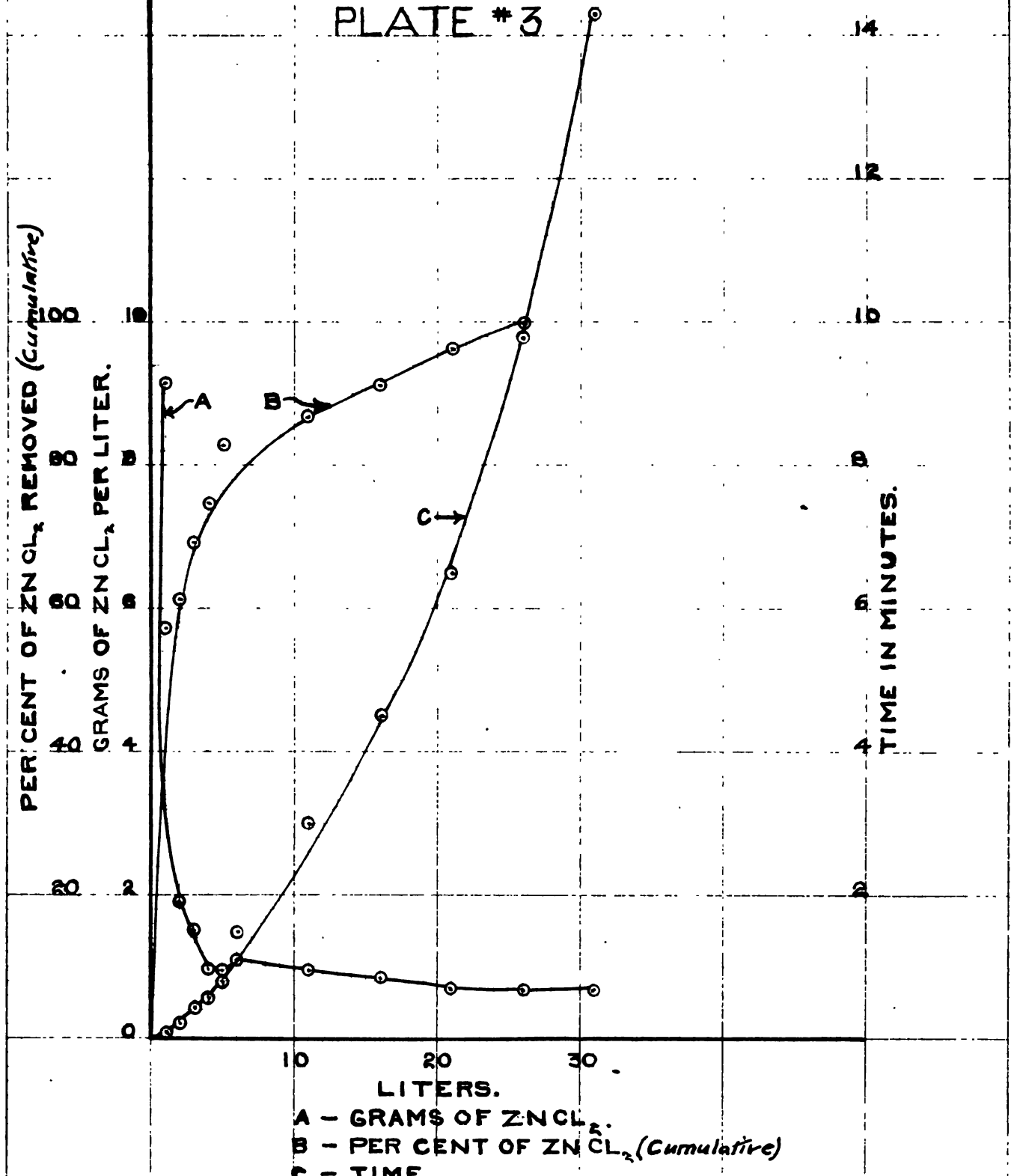
Air pressure of 45 pounds was used.

Liter samples up to # 6. From #7 on samples represent 5 liters.

Sample	Vol. of Sample.	Time	c.c. of N/10 $AgNO_3$.	Gms. of $ZnCl_2$/liter.	% of $ZnCl_2$.
1	50 in 500	10 sec.	6.7	9.15	50.75
2	"	7 "	1.4	1.91	10.60
3	"	5 "	1.1	1.50	8.33
4	"	8 "	0.7	0.955	5.30
5	"	20 "	0.7	0.955	5.30
6	"	37 "	0.8	1.09	6.05
7	" 1 min.	40 sec.	0.7	0.955	5.30
8	" 1 "	30 "	0.6	0.82	4.55
9	" 1 "	50 "	0.5	0.68	3.78
10	" 3 "	20 "	0.5	0.68	(a)
11	" 4 "	30 "	0.5	0.68	
12	" 5 "	5 "	0.5	0.68	

(a) Total to here was called one hundred per cent.

PLATE #3



A – GRAMS OF ZN CL$_2$.
B – PER CENT OF ZN CL$_2$ (Cumulative)
C – TIME.

Test # 4.

Two # 6 batteries were treated in the usual manner.

Thickness of dry cake, 1 inch.

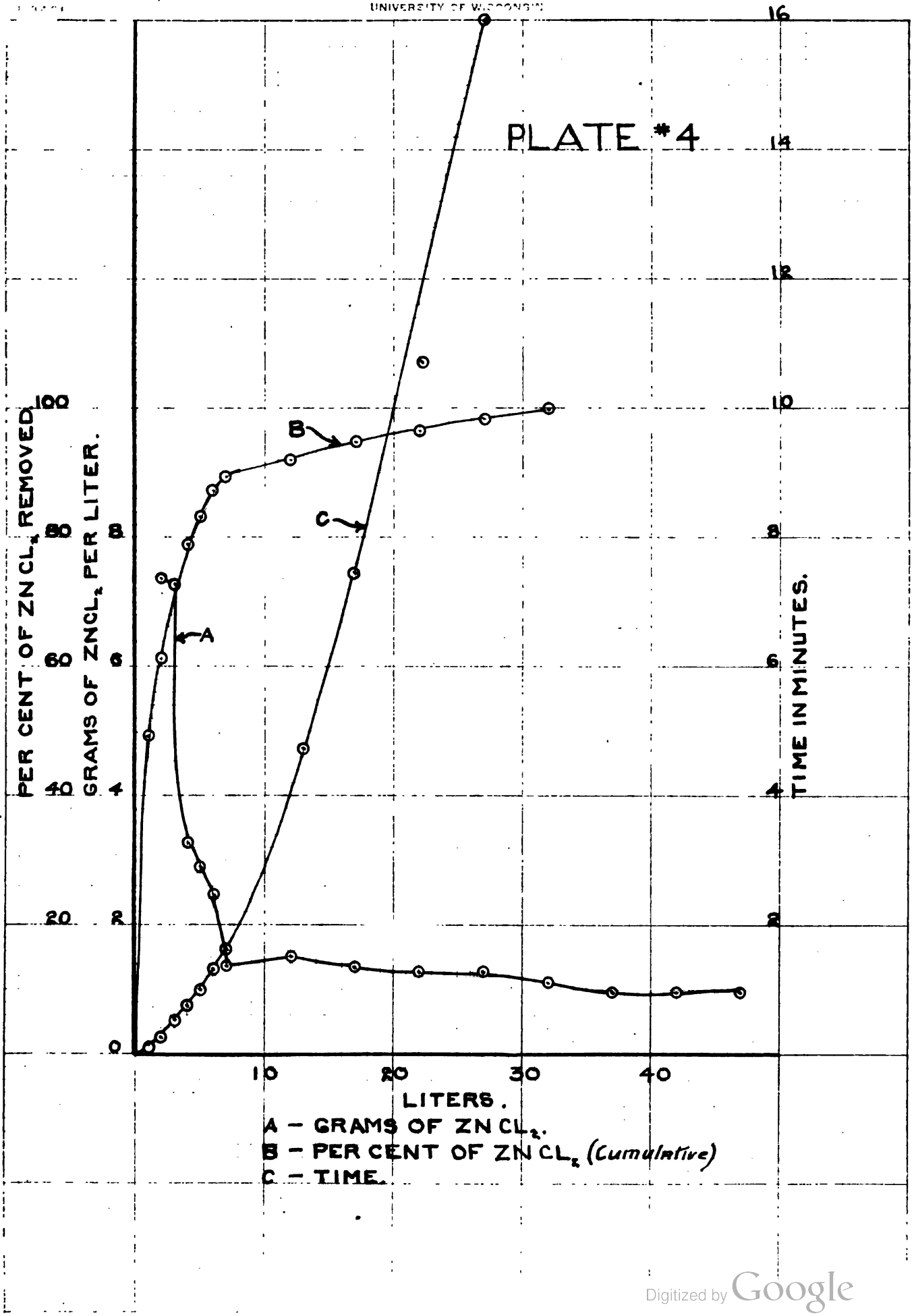
Air pressure of 45 pounds was used.

Liter samples up to # 7. From # 8 on samples represent 5 liters.

Sample	Vol. of Sample	Time	c.c. of N/10 $AgNo_3$.	Gms. of $ZnCl_2$/liter	% of $ZnCl_2$.
1	50 in 500	8 sec.	22	30	49.3
2	"	6 "	5.4	7.35	12.05
3	"	21 "	5.3	7.24	11.9
4	"	12 "	2.4	3.27	5.37
5	"	16 "	2.1	2.87	4.71
6	"	17 "	1.8	2.45	4.03
7	"	17 "	1.	1.36	2.24
8	" 2 min.	10 sec.	1.1	1.50	2.46
9	" 2 "	40 "	1.0	1.36	2.24
10	" 3 "	20 "	0.9	1.23	2.02
11	" 5 "	15 "	0.9	1.23	2.02
12	" 4 "	26 "	0.8	1.09	3.2
13	" 5 "	30 "	0.7	0.95	(a)
14	" 6 "	0 "	0.7	0.95	
15	" 9 "	0 "	0.6	0.95	

(a) Total to here was called one hundred per cent.

PLATE #4



A – GRAMS OF ZN CL₂.
B – PER CENT OF ZN CL₂ (Cumulative)
C – TIME.

Test # 5.

Three # 6 batteries were treated in the usual manner.

Air pressure of 45 pounds was used up to # 8 when 85 pounds water pressure was turned on.

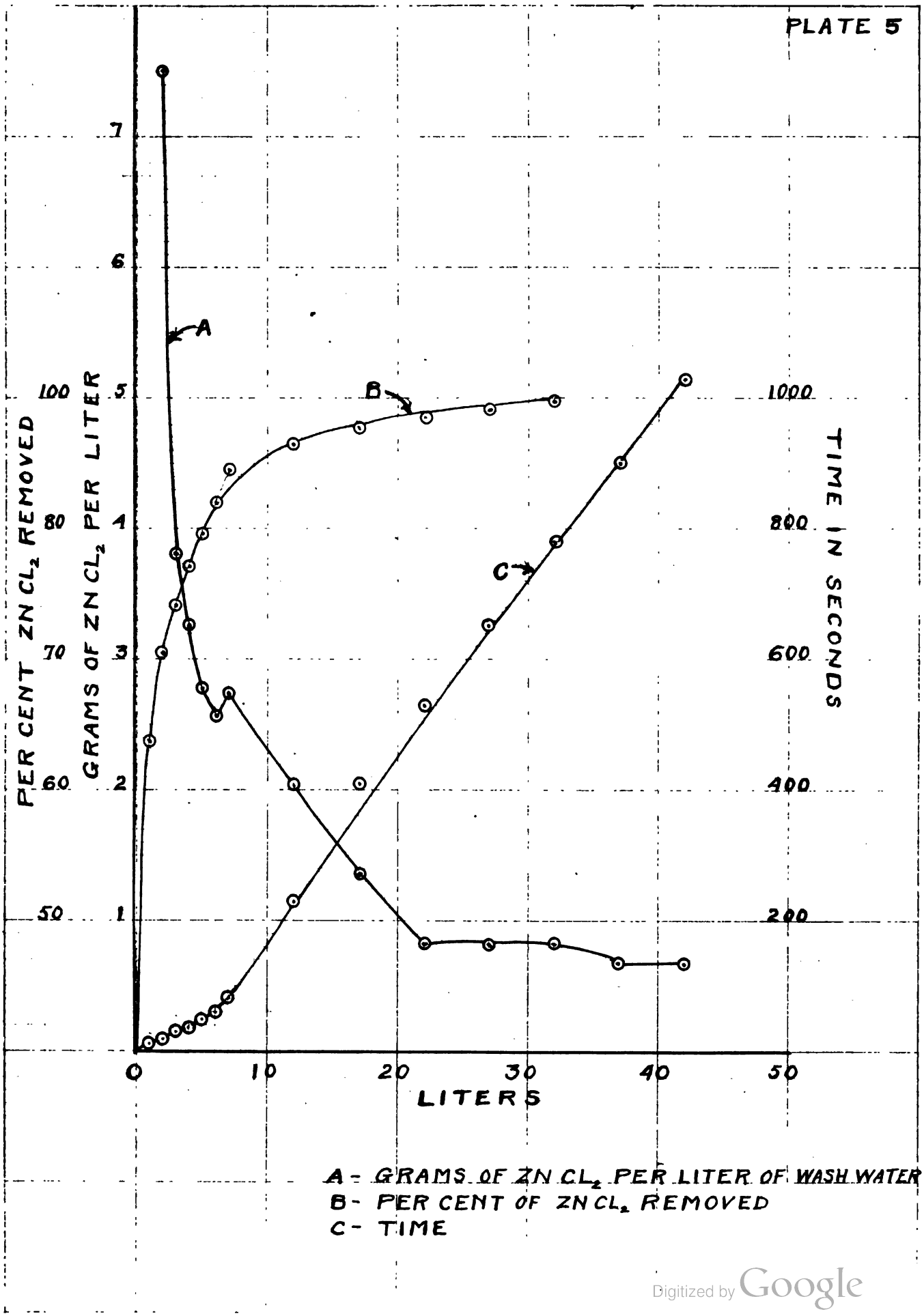
Liter samples up to # 7. From # 8 on samples are 5 liters.

Thickness of cake was not taken.

Sample	Vol. of Sample	Time	c.c. of $AgNO_3$.	Gms. of $ZnCl_2$/liter.	% of $ZnCl_2$.
1	50 in 500	12 sec.	19	25.8	47.5
2	"	8 "	5.5	7.50	13.8
3	"	8 "	2.8	3.81	7.01
4	"	8 "	2.4	3.27	6.02
5	"	12 "	2.0	2.78	5.11
6	"	13 "	1.9	2.58	4.75
7	"	23 "	2.0	2.73	5.03
8	"	2 min.25 sec.	1.5	2.03	3.74
9	"	3 " 0 "	1.0	1.36	2.50
10	"	2 " 0 "	0.6	0.82	1.51
11	"	2 " 4 "	0.6	0.82	1.51
12	"	2 " 5 "	0.5	0.82	1.51
13	"	2 " 0 "	0.5	0.68	(a)
14	"	2 " 2 "	0.5	0.68	
15	"	2 " 4 "	0.5	0.68	
16	"	2 " 5 "	0.5	0.68	

(a) Total to here was called one hundred per cent.

PLATE 5



A - GRAMS OF ZN CL$_2$ PER LITER OF WASH WATER
B - PER CENT OF ZN CL$_2$ REMOVED
C - TIME

Test # 6.

Thickness of cake, dry, 1 7/8 inches.

Water pressure, 85 pounds.

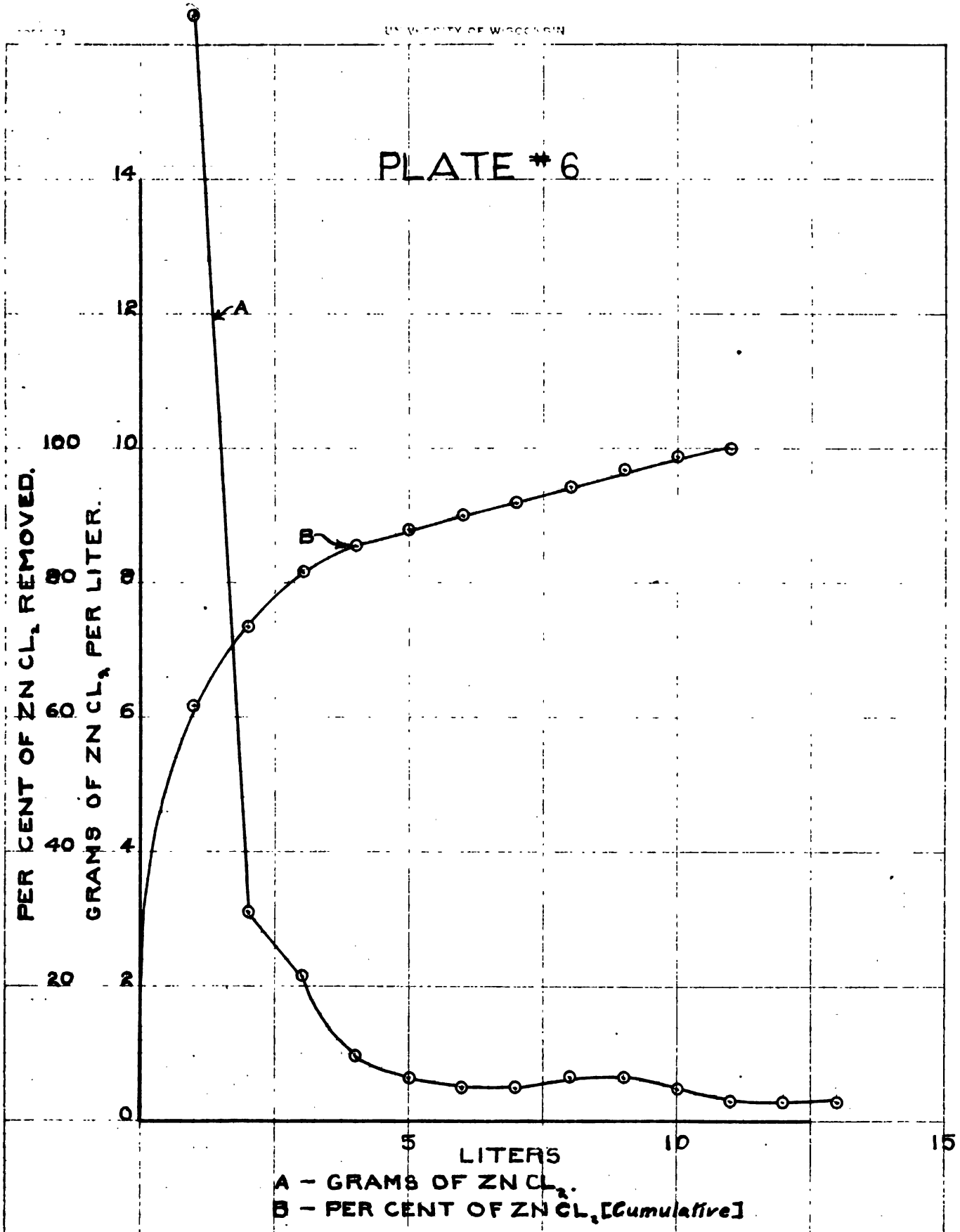
No time was taken.

Samples were taken every liter.

Sample	Vol. of Sample.	c.c. of N/10 $AgNO_3$.	Gms. of $ZnCl_2$/liter.	% of $ZnCl_2$.
1	50 in 500	15.4	16.43	62.2
2	"	2.9	11.70	11.70
3	"	2.0	2.13	8.05
4	"	0.9	0.96	3.63
5	"	0.6	0.64	2.42
6	"	0.5	0.534	2.02
7	"	0.5	0.534	2.02
8	"	0.6	0.64	2.42
9	"	0.6	0.64	2.42
10	"	0.5	0.534	2.02
11	"	0.3	0.32	1.21
12	"	0.3	0.32	(a)
13	"	0.3	0.32	
14	"	0.3	0.32	
15	"	0.3	0.32	
16	"	0.3	0.32	

(a) Total to here was called one hundred per cent.

PLATE * 6



Test #7

Thickness of cake, dry, 1 3/4 inches.

Water pressure,85 pounds.

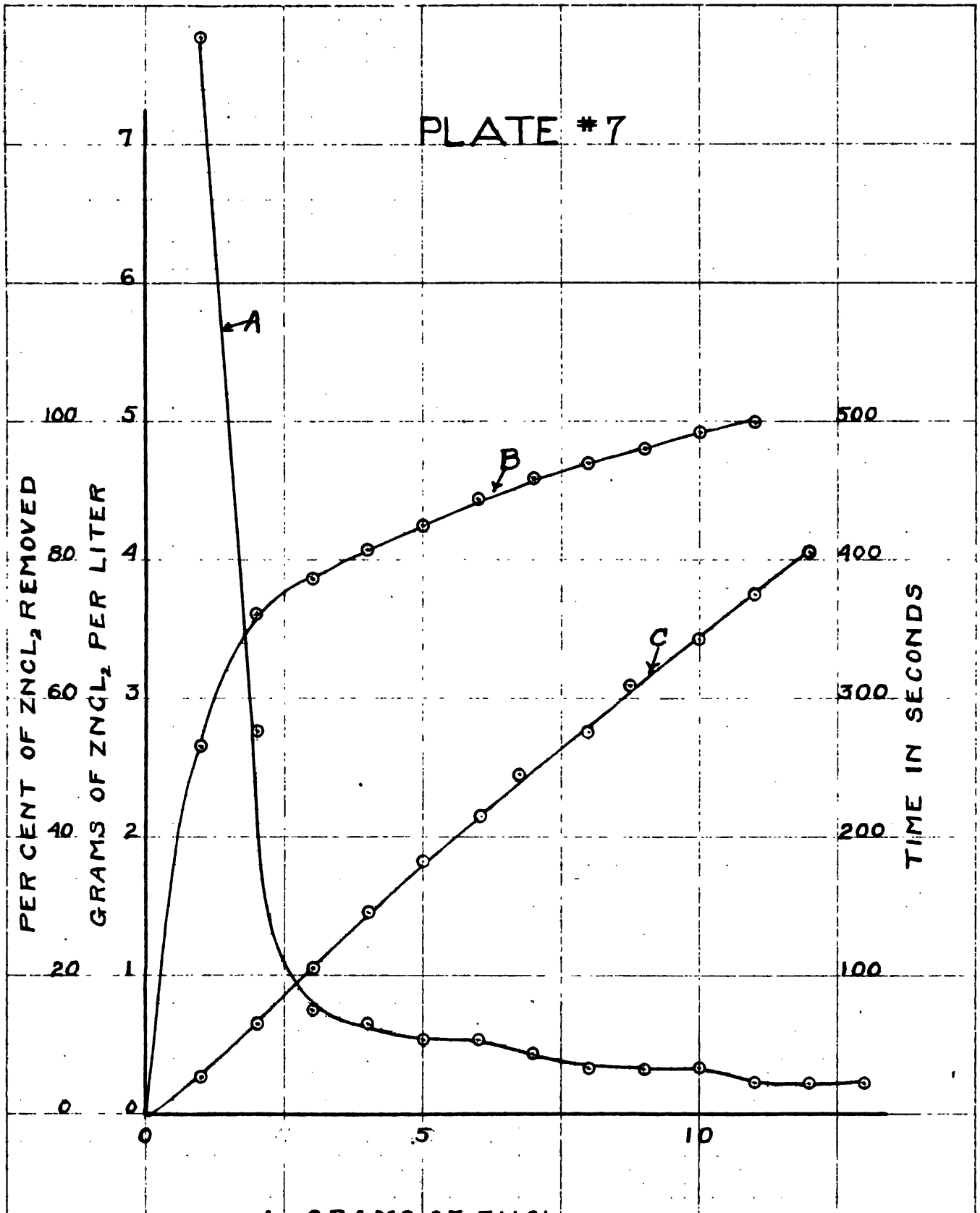
Total time of run, seven minutes and fifty four seconds.

Samples were taken every liter.

Sample	Vol. of Sample	Time in Seconds.	c.c.of N/10 $AgNO_3$	Gms. of $ZnCl_2$/liter.	% of $ZnCl_2$.
1	50 in 500	28	7.3	7.79	53.3
2	"	37	2.6	2.77	18.91
3	"	40	0.7	0.746	5.10
4	"	40	0.6	0.64	4.375
5	"	37	0.5	0.534	3.65
6	"	32	0.5	0.534	3.65
7	"	32	0.4	0.426	2.91
8	"	32	0.3	0.320	2.19
9	"	32	0.3	0.320	2.19
10	"	32	0.3	0.320	2.19
11	"	32	0.2	0.213	1.46
12	"	32	0.2	0.213	(a)
13	"	33	0.2	0.213	
14	"	35	0.2	0.213	

(a) total to here was called one hundred per cent.

PLATE #7



A- GRAMS OF $ZNCL_2$
B- PER CENT OF $ZNCL_2$ (Cumulative)
C- TIME (Cumulative)

Test # 8.

Thickness of cake, dry, 3 1/2 inches.

Water pressure, 85 pounds.

Total time of run, 14 minutes and 23 seconds.

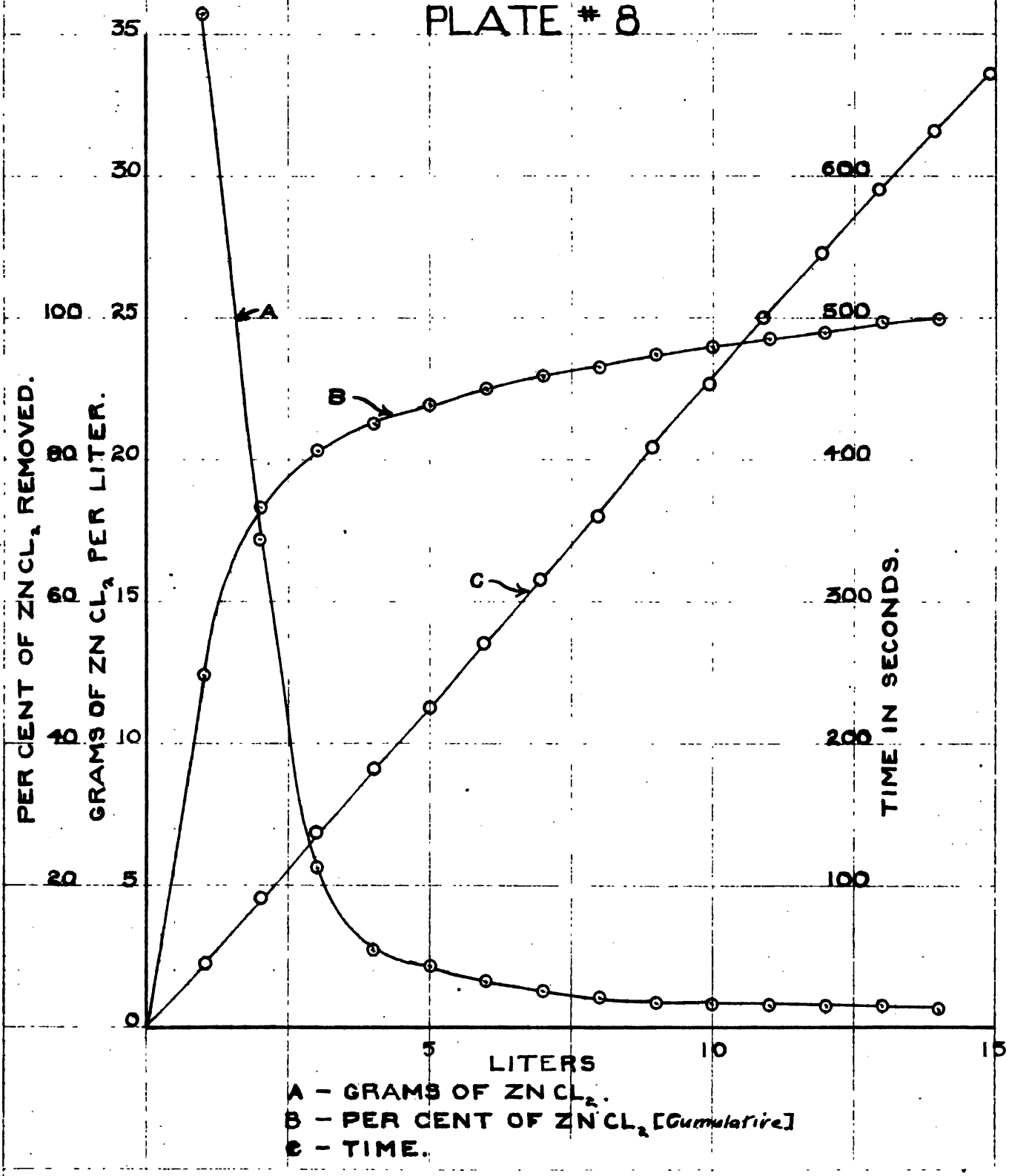
Samples were taken every liter.

Sample	Vol. of Sample.	Time.	c.c. of N/10 $AgNO_3$	Gms. of $ZnCl_2$/liter.	% of $ZnCl_2$.
1	50 in 500	(a)	33.6	35.8	49.6
2	"		16.1	17.19	23.81
3	"		5.3	5.65	7.85
4	"		2.6	2.67	3.70
5	"		2.0	2.13	2.96
6	"		1.5	1.60	2.22
7	"	46 sec.	1.2	1.28	1.77
8	"	46 "	1.0	1.06	1.47
9	"	46 "	0.9	0.96	1.33
10	"	46 "	0.8	0.854	1.18
11	"	46 "	0.7	0.746	1.03
12	"	46 "	0.7	0.746	1.03
13	"	46 "	0.7	0.746	1.03
14	"	42 "	0.6	0.640	0.889
15	"	40 "	0.6	0.640	(a')
16	"	40 "	0.6	0.640	
17	"	37 "	0.6	0.640	
18	"	30 "	0.6	0.640	

(a')Total to here was called one hundred per cent.

(a) Due to trouble with the press no time was taken up to # 7.

PLATE # 8



A – GRAMS OF ZN CL_2.
B – PER CENT OF ZN CL_2 [Cumulative]
C – TIME.

Test # 9.

Thickness of cake, dry, was 4 inches.

Water pressure of 85 pounds was used.

Samples were taken every liter.

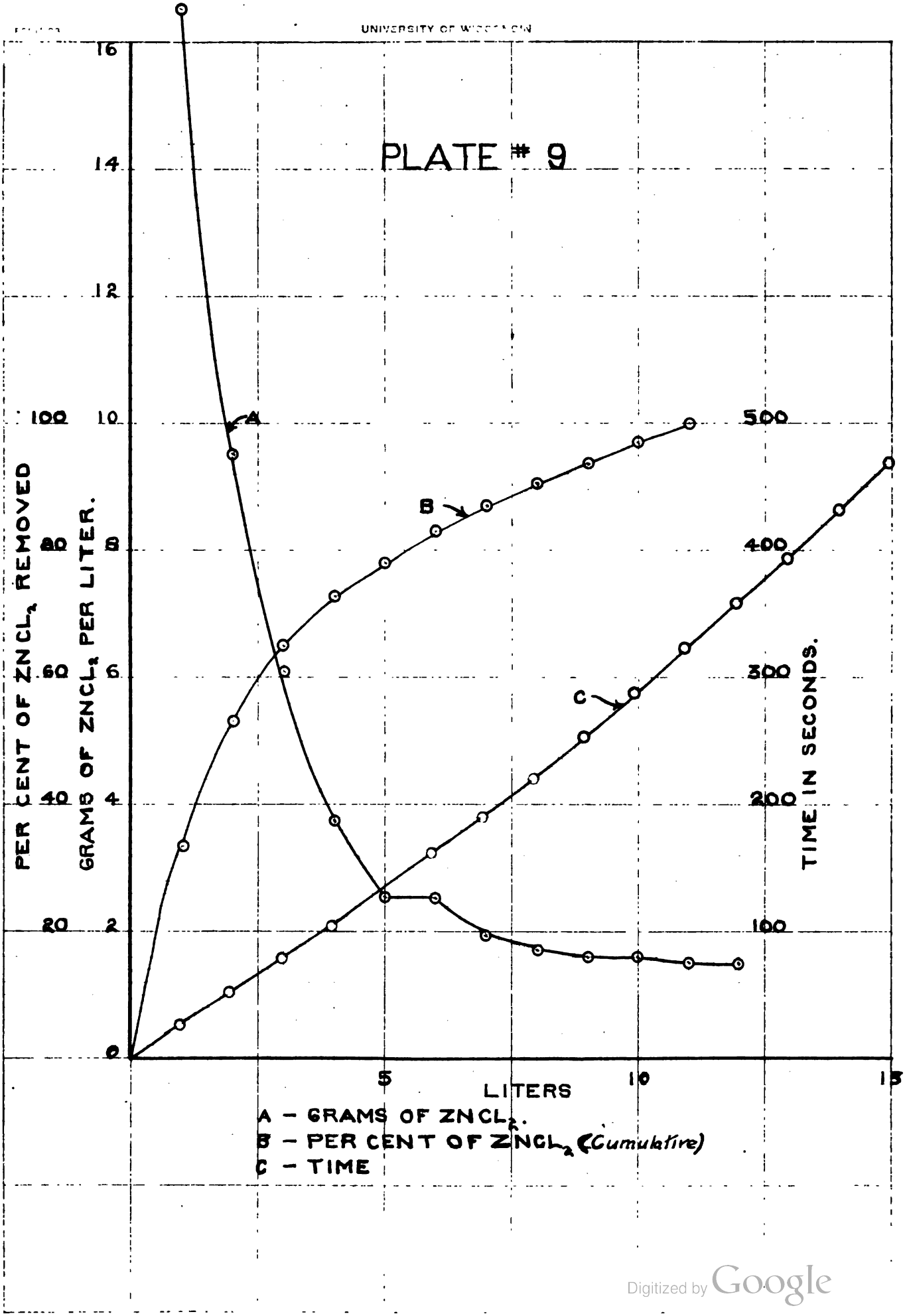
Batteries were leached before using.

Total time of run was 3 minutes and 56 seconds.

Sample	Vol. of Sample.	Time	c.c. of $AgNO_3$.	Gms. of $ZnCl_2$/liter	% of $ZnCl_2$.
1	50 in 500	12 sec.	15.5	16.53	33.6
2	"	14 "	8.9	9.50	19.3
3	"	13 "	5.70	6.08	12.3
4	"	14 "	3.5	3.73	7.6
5	"	14 "	2.4	2.56	5.2
6	"	14 "	2.4	2.56	5.2
7	"	14 "	1.8	1.92	3.9
8	"	15 "	1.6	1.70	3.45
9	"	17 "	1.5	1.60	3.24
10	"	17 "	1.5	1.60	3.24
11	"	18 "	1.4	1.49	3.03
12	"	18 "	1.4	1.49	(a)
13	"	18 "	1.4	1.49	
14	"	19 "	1.4	1.49	
15	"	19 "	1.4	1.49	
16	"	19 "	1.4	1.49	
17	"	20 "	1.4	1.49	
18	"	20 "	1.4	1.49	

(a) Total to here was called one hundred per cent.

PLATE # 9



Test # 10.

Thickness of cake, dry, was 6 1/4 inches.

Batteries were leached two days before using.

Total time of run was 14 minutes and 10 seconds.

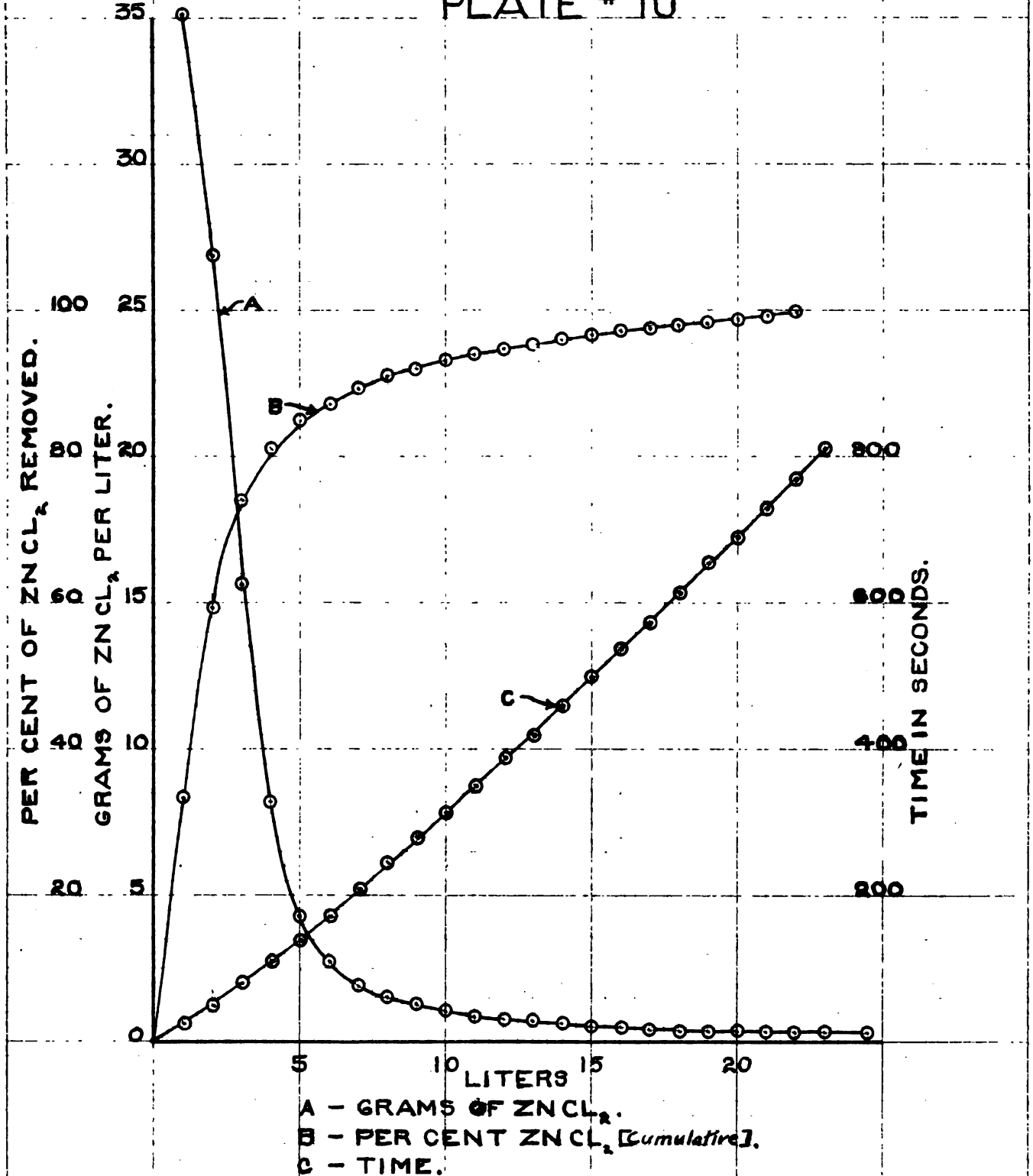
Water pressure of 85 pounds was used.

Samples were taken every liter.

Sample	Vol. of Sample.	Seconds Time	c.c.of $AgNO_3$.	Gms.of $ZnCl_2$/liter.	% of $ZnCl_2$.
1	10/liter	23	51.4	35.1	33.6
2	"	27	39.4	26.9	25.7
3	"	30	22.9	15.6	14.9
4	"	30	11.9	8.13	7.8
5	"	32	6.3	4.3	4.1
6	"	34	3.95	2.695	2.6
7	"	35	2.9	1.98	1.9
8	"	35	2.2	1.5	1.4
9	"	35	1.8	1.229	1.17
10	"	35	1.6	1.091	1.04
11	"	36	1.3	0.887	0.85
12	"	36	1.1	0.751	0.72
13	"	37	1.0	0.6825	0.658
14	"	38	0.9	0.614	0.587
15	"	38	0.8	0.546	0.522
16	"	38	0.8	0.546	0.5222
17	"	39	0.7	0.478	0.457
18	"	39	0.6	0.409	0.391
19	"	39	0.6	0.409	0.391
20	"	39	0.6	0.409	0.391
21	"	39	0.5	0.342	0.327 (a)

(a)Total to here was called one hundred per cent.

PLATE # 10



Test # 11.

The ingredients of two # 8 dry batteries, volume about 147 cubic inches, after being digested with a solution of ten per cent caustic soda, were allowed to stand in five gallons of water for two days and then were put through the filter press in the regular manner.

Thickness of cake, dry, 1-1/4 inches.

Water pressure of 85 pounds was used.

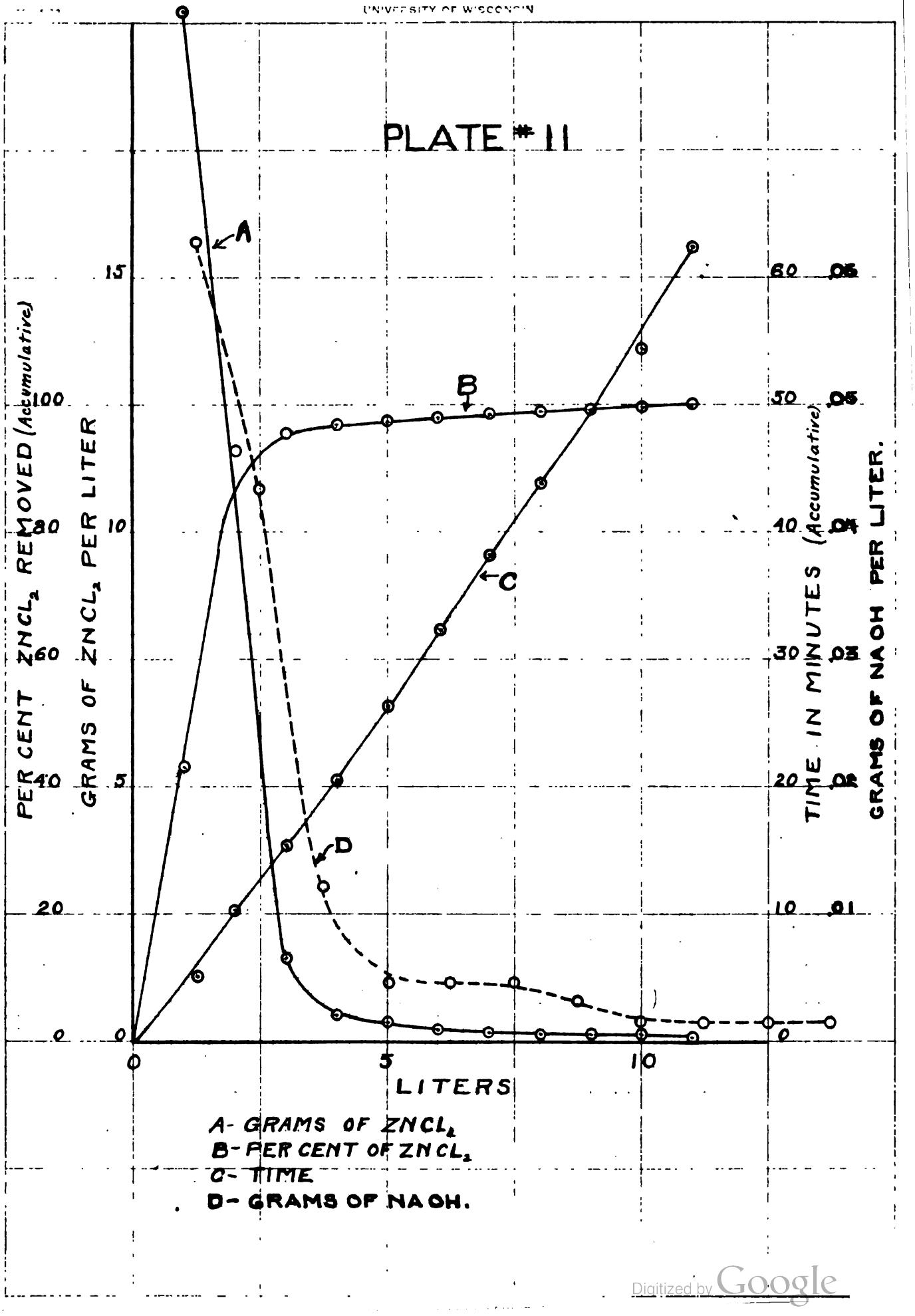
Samples were taken every liter.

Total time of the run was 1 hour and 1 minute.

Samples for analysis are 10 c.c. per liter.

Sample	Time	c.c. of .75N H_2SO_4	Grams of NaOH/liter	c.c. of N/10 $AgNO_3$	Gms. of NaCl/liter	Perc't NaCl
1	5 min. 10 sec.	2.1	.063	34.6	20.23	42.9
2	5 " 5 "	1.45	.0435	40.0	23.41	49.6
3	5 " 0 "	0.4	.012	2.8	1.64	3.48
4	5 " 25 "	0.15	.0045	0.9	0.5260	1.12
5	5 " 45 "	0.15	.0045	0.6	0.3513	0.74
6	6 " 0 "	0.15	.0045	0.4	0.2340	0.50
7	5 " 45 "	0.10	.0030	0.3	0.1757	0.37
8	5 " 45 "	0.05	.0015	0.3	0.1757	0.37
9	5 " 30 "	0.05	.0015	0.3	0.1757	0.37
10	6 " 0 "	0.05	.0015	0.3	0.1757	0.37
11	5 " 50 "	0.05	.0015	0.25	0.1462	0.31

PLATE # 11



Test # 12.

The ingredients of two # 8 dry batteries, volume about 147 cubic inches, were leached for two days in five gallons of water and then put through the digestor as in the previous experiment. The mixture from the digestor was again leached for some time and then run through the filter press in the regular manner.

Thickness of cake, dry, 1-1/2 inches.

Water pressure of 85 pounds was used.

Samples were taken every liter.

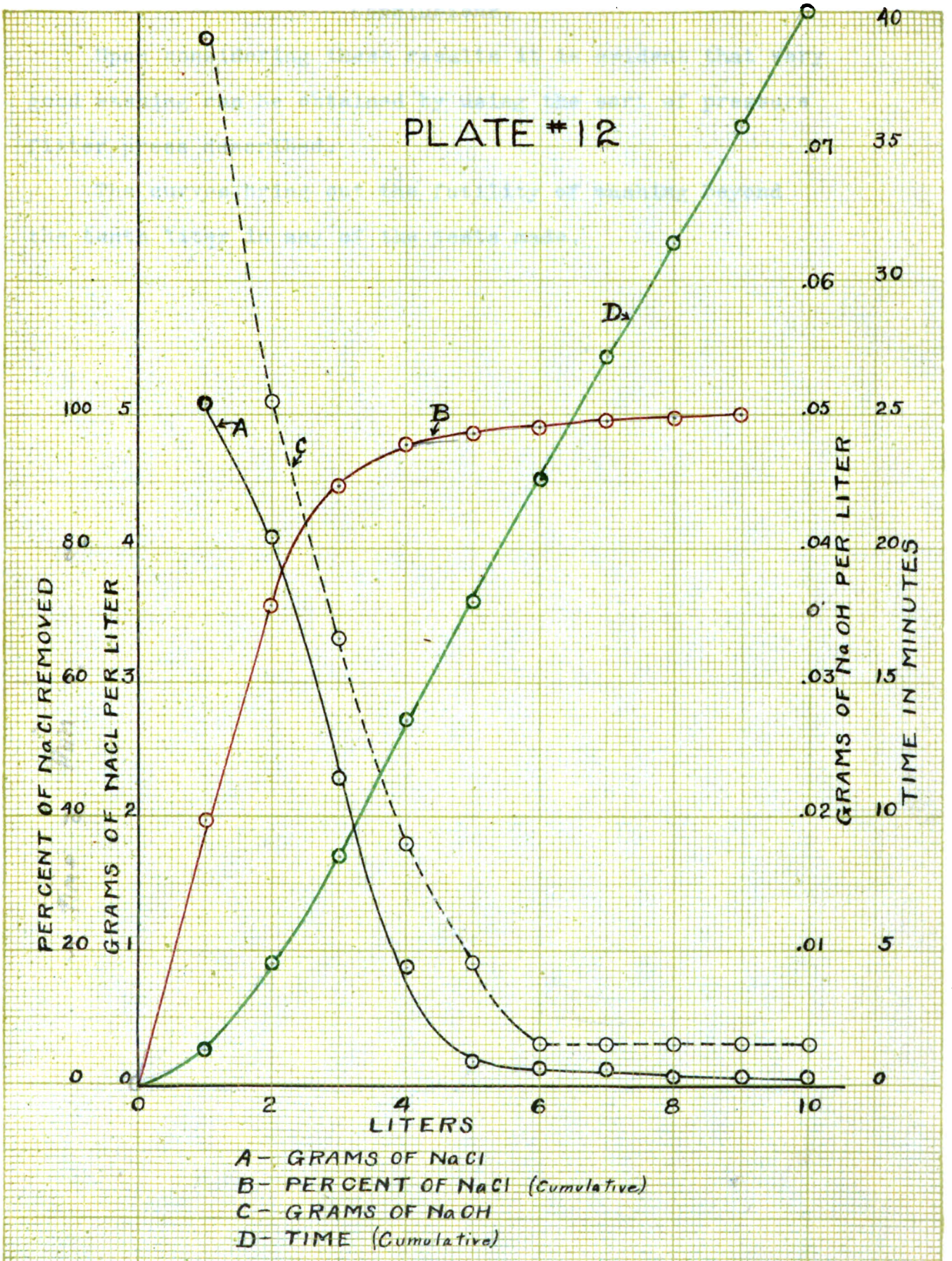
Total time of the run was 40 minutes and 6 seconds.

Samples for analysis are 10 c.c. per liter.

The material was digested for about 5 hours.

Sample	Time	c.c. of .75 N H_2SO_4	Gms. of NaOH/liter	c.c. of $AgNO_3$	Gms. of NaCl/liter	Perc't NaCl
1	1 min 20 sec.	2.6	0.078	8.7	5.09	39.6
2	3 " 11 "	1.7	0.051	7.0	4.09	31.81
3	4 " 10 "	1.1	0.033	3.9	2.28	17.71
4	5 " 0 "	0.6	0.018	1.5	0.878	6.82
5	4 " 20 "	0.3	0.009	0.3	0.1755	1.36
6	4 " 40 "	0.1	0.003	0.2	0.1170	0.91
7	4 " 30 "	0.1	0.003	0.2	0.1170	0.91
8	4 " 15 "	0.1	0.003	0.1	0.0585	0.45
9	4 " 20 "	0.1	0.003	0.1	0.0585	0.45
10	4 " 20 "	0.1	0.003	0.1	0.0585	0.45

For titration a volume of silver nitrate was made up, 1 c.c. of which was equivalent to .005853 grams of NaCl. This was also used in test # 11.



Conclusions.

Upon considering these results it is evident that very good washing can be obtained by using the sort of pressure filter press described.

The curves bring out the futility of washing beyond the tenth liter in any of the tests made.

Approved,

T. Howard

June 8, 1915.